

**-NANOSTRUCTURES AND ENHANCED PROPERTIES IN TUNGSTEN AND ITS
ALLOYS PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING**

Interim Report No. 0003

by

**R.Z. Valiev and I.V. Alexandrov
(11 October 2001 - 10 January 2002)**

United States Army

EUROPEN RESEARCH OFFICE OF THE U.S. ARMY

London England

CONTRACT NUMBER: N68171-01-M-5641

9135-MS-01

Name of Contractor

**Institute of Physics of Advanced Materials
Ufa State Aviation Technical University
Russia**

Approved for Public Release; Distribution unlimited

INTERIM REPORT

20020215 136

A. The Cover Page

- (1) Nanostructure and enhanced properties in tungsten and its alloys processed by equal channel angular pressing.
- (2) Professor R.Z. Valiev
- (3) Professor V.S. Zhernakov
- (4) N68171-01-M-5641
- (5) Third Interim Report
- (6) 11 October 2001 - 10 January 2002
- (7) The Research reported in this document has been made possible through the support and sponsorship of the U.S. Army. This report is intended only for the internal management use of the Contractor and U.S. Government.

The results of the investigation carried out in the frame of the first two periods of the present contract N6871-01-M-5641 have shown, that severe plastic deformation (SPD) by means of equal-channel angular pressing (ECAP), being an effective way of microstructure refinement and enhancement of mechanical properties of metals and alloys can be successfully applied for hard-to-deform and low-ductility W and the 90W-8Ni-2Fe alloy. However, achieving the minimum grain size less than 0.5 μm by ECAP as well as obtaining homogeneous ultrafine grained structure because of low workability of these materials are still rather problematic.

In this connection, in the frame of the third period of the present project there have been conducted experimental investigations, aimed at ECAP optimization of the investigated W and the 90W-8Ni-2Fe alloy. The problem of this task consists in enhancing workability of these hard-to-deform materials and achieving large strains. The influence of the accumulated strain degree, the thickness of protection casing, the rate and strain temperatures on grain size and homogeneity of the obtained structure have been investigated as parameters, determining ECAP results. ECAP has been realised on route C, which turns out to be the most effective route for the given case, as it has been found out at first stages of the project.

One of the main results of the investigations conducted for the reported period on the example of CP W, is achieving the accumulated strain degree $\epsilon \approx 8$. It could take place at the expense of reducing the accumulated strain degree for 1 pass down to 0.5, and also by increasing the overall number of passes up to 16 at the expense of applying the dies with an angle intersection equal to 110° and 135°.

The results of microstructure investigation shows, that billets after 4 passes of ECA pressing at a rate of 6 and 300 mm/s have approximately the same average grain size (about 0.6 μm). At the same time, ECAP rate increasing up to 300 mm/s have not provided the enough homogeneous treatment of structure. It can be perfectly seen at optical microstructure pictures. TEM structure along with equiaxed grains have also shown a large share of elongated grains and subgrains.

With increasing the number of cycles and reducing ECAP rate the slightly deformed microstructure areas have been practically disappearing and the share of equiaxed grains and subgrains up to 75÷80% of the volume after 8 ECAP passes have been increasing. Metallographic texture, typical for the initial state, is disappearing along with increasing the accumulated strain rate. After ECAP with 10 passes the average grain size has turned out to be less than 0.4 μm . The corresponding microhardness has increased from 5020 MPa to 6490 MPa.

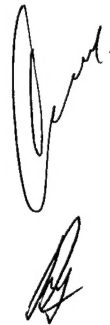
Approaches, worked out during ECAP of CP W, have been applied to refine microstructure in the 90W-8Ni-2Fe alloy. It has been managed to carry out 3 successful passes without the billet destruction. Optical metallography shows that grains of W phase have been fragmented. TEM investigations testify to the fact, that the substructure with small angle dislocation boundaries, having dimensions around 1 μm is forming in W phase during ECAP. At the same time W microhardness (α -phase) after ECAP has increased from 5550 MPa to 6580 MPa, microhardness of interphase layer (γ -phase) has increased from 4210 MPa to 5320 MPa, i.e. microhardness of both phases has increased by more than 1000 MPa.

However it should be mentioned, that the problem of obtaining integral bulk billets having proper cylinder form still remains topical, in spite of the fact that the tasks (put forward in the project) to refine the average grain size in CP W up to dimensions less than 0.5 μm and to enhance strength characteristics have been successfully achieved. The above mentioned problem is connected with decreasing the temperature of the billet during ECAP and changing the character of the material flow. That is why it is necessary to approximate ECAP conditions to the isothermal ones. In this connection there has been started fabrication of the die from the heat-resistant (up to 900°) Ni-based superalloy during the reported period.

The use of the given die during the 4th period of the project execution will allow to conduct ECAP in conditions, close to the isothermal ones. The use of fluid glass lubrication will make it possible to replace metallic protective coverings and to guarantee high tribological characteristics during ECAP and the protection of billets from oxidation at high temperatures. At the same time it allows to obtain the deformed billets with geometric dimensions, close to the initial ones. Taking into account high working pressures and temperatures, the main difficulty in this case is to provide high structural strength of the die at ECAP temperatures.

Apart from these tasks, in the frame of the 4th final period of the present project there will be conducted complex investigations of mechanical properties of CP W and the 90W-8Ni-2Fe alloy after ECAP for establishing the influence of ultrafine grained structures on the temperature of the brittle-ductile transition.

On the basis of the conducted investigation results there have been prepared a paper conjointly with Dr. Robert J. Dowding (U.S. Army Research Laboratory) for the International Conference on Refractory & Hardmetals 2002 (London). There will be also prepared and presented a joint report at the Second International Symposium on Ultrafine Grained Materials, which is to be held in the frame of 2002 TMS Annual Meeting from 17 to 21 February, 2002 in Seattle (USA). A joint article will be published in the proceedings of this Symposium as well.



REPORT DOCUMENT PAGE			<i>Form Approved</i> <i>OMB No 0704</i>	
1. AGENCY USE ONLY.		2. REPORT DATE January 10, 2001	3. REPORT TYPE AND DATES III Interim Report (11.10.2001 - 10.01.2002)	
4. TITLE AND SUBTITLE Nanostructures and enhanced properties in Tungsten and its alloys processed by equal channel angular pressing			5. FUNDING NUMBERS N68171-01-M-5641	
6. AUTHORS: Professor R.Z. Valiev and Dr. I.V. Alexandrov				
7. PERFORMING ORGANIZATION NAMES AND ADDRESS Institute of Physics of Advanced Materials, Ufa State Aviation Technical University. 12 K.Marx str., Ufa 450000, Russia			8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME AND ADDRESS U.S. Government and European Research Office of the U.S. Army. USARDSG UK, FISCAL-OFFICE, DR.ILLINGER, EDISON HOUSE, 223OLD MARYLEBONE ROAD, LONDON NW1 5TH, UNITED KINGDOM			10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES In co-operation with Dr. R. Dowding, U.S. Army Research Laboratory Aberdeen Proving Ground, Maryland 21005-5066				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release, distribution unlimited			12b. DISTRIBUTION CODE	
13. ABSTRACT The given report presents the results of developing the equal channel angular pressing (ECAP) technique for processing hard-to-deform and low-ductility CP W and the 90W-8Ni-2Fe alloy. With the help of specialized dies and determination of optimal pressing regimes it has been managed to increase the accumulated strains in CP W up to $8 \div 10$ and at this expense to obtain the average grain size less than $0.5 \mu\text{m}$ and microhardness 6490 MPa. The 90W-8Ni-2Fe alloy has been successfully subjected to ECAP with 3 passes. There has been found out a difference in microstructure refinement of W phase (α -phase) and γ Ni-based solid solution (γ -phase).				
14. SUBJECT TERMS Tungsten and its alloys, equal channel angular pressing, Ultrafine-grained microstructure			15. NUMBER OF PAGES	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL	